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Patents Form 1/77

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31OCT02 E759939-1 002819
P01/7700 0.00-0225329.2

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

The Patent Office

Cardiff Road
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South Wales
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1. Your reference C438/G

2. Patent application number *(The Patent Office will fill in this part)* 0225329.2 31 OCT 2002

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Genevac Ltd
Sovereign Centre
Farthing Road
Ipswich
IP1 5AP

Patents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation England

4. Title of the invention

Pressure control in Centrifugal Evaporators

5. Name of your agent *(if you have one)* Keith W Nash & Co

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

90-92 Regent Street
Cambridge
CB2 1DP

Patents ADP number *(if you know it)* 1206001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day / month / year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d)) Yes

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form

Description 6

Claim(s)

Abstract

Drawing(s) 3 + 3

CF

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*

Request for preliminary examination and search *(Patents Form 9/77)*

Request for substantive examination *(Patents Form 10/77)*

Any other documents *(please specify)*

11. I/We request the grant of a patent on the basis of this application.

Signature Keith W Nash Date 30/10/2002

Keith W Nash & Co., Agent

12. Name and daytime telephone number of person to contact in the United Kingdom

Mr Nash 01223 355477

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Notes

a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 08459 500505.

b) Write your answers in capital letters using black ink or you may type them.

c) If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.

d) If you have answered 'Yes' Patents Form 7/77 will need to be filed.

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C438/G

Title: Pressure control in Centrifugal Evaporators

Field of invention

This invention concerns drying apparatus, especially centrifugal evaporators and in particular the control of pressure within the drying chamber of such apparatus.

Background

Control of pressure in drying apparatus such as centrifugal evaporators is important for 3 reasons:

(i) To allow controlled reduction of the chamber pressure in order to prevent bumping

(ii) To limit the ultimate chamber pressure so as to prevent solvent from freezing during evaporation. This is particularly important when evaporating water. Evaporation from the solid phase (sublimation) is much slower than evaporation from the liquid phase.

(iii) To limit the ultimate chamber pressure in order to maximise solvent recovery. This is particularly important with volatile solvents, typically those that would boil at –30°C or below at the ultimate pressure achieved in the chamber.

There are two methods in common use for controlling pressure:

Figure 1 of the accompanying drawings illustrates a first method which has been used, possibly for 20 years or more to control pressure in Freeze Dryers. This method involves connecting an adjustable bleed valve to the drying chamber and steadily bleeding a controlled amount of nitrogen or air into the chamber while continuously vacuum pumping the chamber. This method allows objectives (ii) and (iii) to be achieved but objective (i)

proved very difficult to achieve when the object is to secure an increasing vacuum ramp. Proportional control of the bleed valve over a large dynamic range would be required to achieve all three requirements listed above.

A second method is described in the US Patent 5,137,604 as particularly shown in Fig 2. Here the pump is isolated from the chamber in response to a signal proportional to the pressure within the drying chamber. This method works well with pumps that: a) do not suffer from condensation of vapours internally (e.g. scroll pumps, vane pumps with gas ballasting), b) would shed oil if operated open to atmosphere for extended periods (vane pumps). This method has some disadvantages: the isolation valve must be solvent resistant and as such is a relatively expensive component because the bore must be large. When used to control the pressure with pumps that suffer from performance loss when solvents condense internally it is found that performance decreases significantly over the duration of an evaporation run. Pump performance is only recovered when a significant flow rate of gas is allowed through the pump to force out the condensed solvent. Multi-head diaphragm pumps, which are commonly used for evaporation equipment are affected badly by solvents condensing internally. An additional problem which has been found due to liquid solvent entrapment within the pump, is premature degradation of the pump components especially elastomeric seals within the pump.

Object

It is an object of the invention to provide an improved method and apparatus for removing a solvent from a mixture in a closed chamber so as to leave a dry residue, in which the pressure within the chamber is very precisely controlled. Apparatus for solvent removal will be referred to as drying apparatus. Drying apparatus can include centrifugal evaporators, freeze driers, and rotary evaporators in which a film of solvent is maintained over the inside surface of a flask by rotating it in a warm fluid.

Summary of the invention

According to the present invention there is provided a method of controlling the pressure within a chamber of a drying apparatus from which air and vapour is removed by a pump which is operated continuously during the drying process, wherein both of a vent valve and a pressure control valve are opened so that air at atmospheric pressure is drawn by the pump directly from the pressure control valve, and via the chamber and a non-return valve from the vent valve and when the pressure in the chamber is to be reduced both vent and pressure control valves are closed to allow the pump to remove air, gas and vapour from the chamber via the non-return valve, and the dropping chamber pressure is monitored by a pressure transducer and after a required chamber pressure has been reached, the pressure control valve is opened while the vent valve remains closed, whereby a high rate of airflow is maintained through the pump to clear the interior of the pump of any residual solvent, and the chamber pressure remains substantially constant until the control valve is closed or partially closed again.

The invention therefore prevents solvent condensation within the pump which can otherwise occur when the pump is isolated from the chamber.

In operation with the vent valve closed and the pressure control valve open, air at atmospheric pressure enters through the pressure control valve and the non-return valve sees atmospheric pressure on the pump side and a "lower" partial (or complete) vacuum pressure in the chamber on the other side. The differential pressure keeps the non-return valve closed, thereby sealing the chamber from atmosphere. However the open pressure control valve provides a ready supply of air at atmospheric pressure to the pump inlet which thus maintains a high flow rate therethrough. This clears any solvent from the interior of the pump.

Clearing solvent from the internal surfaces of the pump ensures that the pump will continue to operate at peak performance and extends the life of the pump in service by eliminating solvent attack.

According to another aspect of the present invention there is provided drying apparatus which includes a drying chamber and means for controlling the pressure within the chamber, in which air gas and vapour is removed from the chamber by a pump which is operated continuously during the drying process, and which further comprises a vent valve to admit air to the chamber, a pressure control valve which when open admits air directly to the pump inlet, a non-return valve between the chamber and the pump inlet, and control means adapted to close both vent and pressure control valves when the pressure in the chamber is to be reduced, the pump removing air, gas and vapour from the chamber through the non-return valve, a pressure transducer adapted to monitor the chamber pressure and to provide a signal to the control means when a required chamber pressure has been reached, the control means being adapted to at least partly open the pressure control valve in response to such a signal while keeping the vent valve closed, whereby a high rate of air flow is maintained through the pump to clear the interior of the pump of solvent, while the chamber pressure is to remain substantially constant, and until the control valve is closed again, and air, gas and vapour from the chamber is once again drawn from the chamber to further reduce the pressure therein.

The pressure control and vent valves can be low cost valves, as they are not subjected to solvent vapour, only air.

The non-return valve may be a flap valve.

The non-return valve does not have to provide a 100% seal, as it is only required to close when the system is maintaining the chamber pressure. If some leakage occurs during this process, the pressure in the chamber will begin to rise and by sensing this and closing (or partially closing) the pressure control valve, the pump is re-connected to the chamber sooner than would have otherwise been the case, to draw air, gas and vapour from the chamber and thereby regain the required lower pressure, whereupon the pressure control valve is once again opened up and the non-return valve again operates to close off the chamber.

Where the non-return valve is a flap valve it can be manufactured in the same way as non-return valves which are used internally within many diaphragm pumps. These valves are low cost but nevertheless solvent resistant, and are "passive" valves in that they close in response to a differential pressure in one direction.

The benefits provided by the invention are:

(a) Pump performance is no longer degraded due to condensation within the pump.

(b) Premature failure of the pump due to internal chemical attack from condensed solvents within the pump is largely eliminated.

(c) The cost of a pressure control system is reduced since it is no longer necessary to use chemically resistant solenoid valves.

The invention will now be described by way of example with reference to Fig 3 of the accompanying drawings.

Fig 3 illustrates a centrifugal evaporator 10 having a sealable chamber 12 containing a sample holding rotor 14 driven by a motor 16. A normally open solenoid operated vent valve 18 controls the admission of air to the chamber.

A pipeline 20 allows air, gas and vapours to be drawn out of the chamber 12 via a condenser 22 having a drain valve 24 and thereafter via a non-return valve 26 provided in accordance with the invention between the trap outlet 28 and a vacuum pump 30. The condenser may be chilled to more effectively condense the mixture leaving the chamber 12.

A second normally open solenoid operated valve 32 allows air at atmospheric pressure to enter the line 34 downstream of the valve 26 in accordance with the invention, to allow air at atmospheric pressure to be available to the inlet to the pump 30 along line 34.

The outlet of pump 30 communicates with atmosphere at 36 possibly via a filter or gas collecting vessel (not shown) if it is not appropriate to allow any of the air/gas from the chamber 12 to exit directly to atmosphere.

A programmable control system supplies control signals to the solenoid operated valves 18, 32, and operating current to the motor 16 and to a motor (not shown) of pump 38. A pressure sensor 40 monitors the chamber pressure by a connection to the condenser 22 and supplies a pressure-indicating signal to 38.

The control system is preferably computer controlled and has appropriate interface boards for converting digital signals from the computer to analogue signals or operating currents as appropriate, and for converting the typically analogue signal from the transducer 49 to a digital signal for use by the computer.

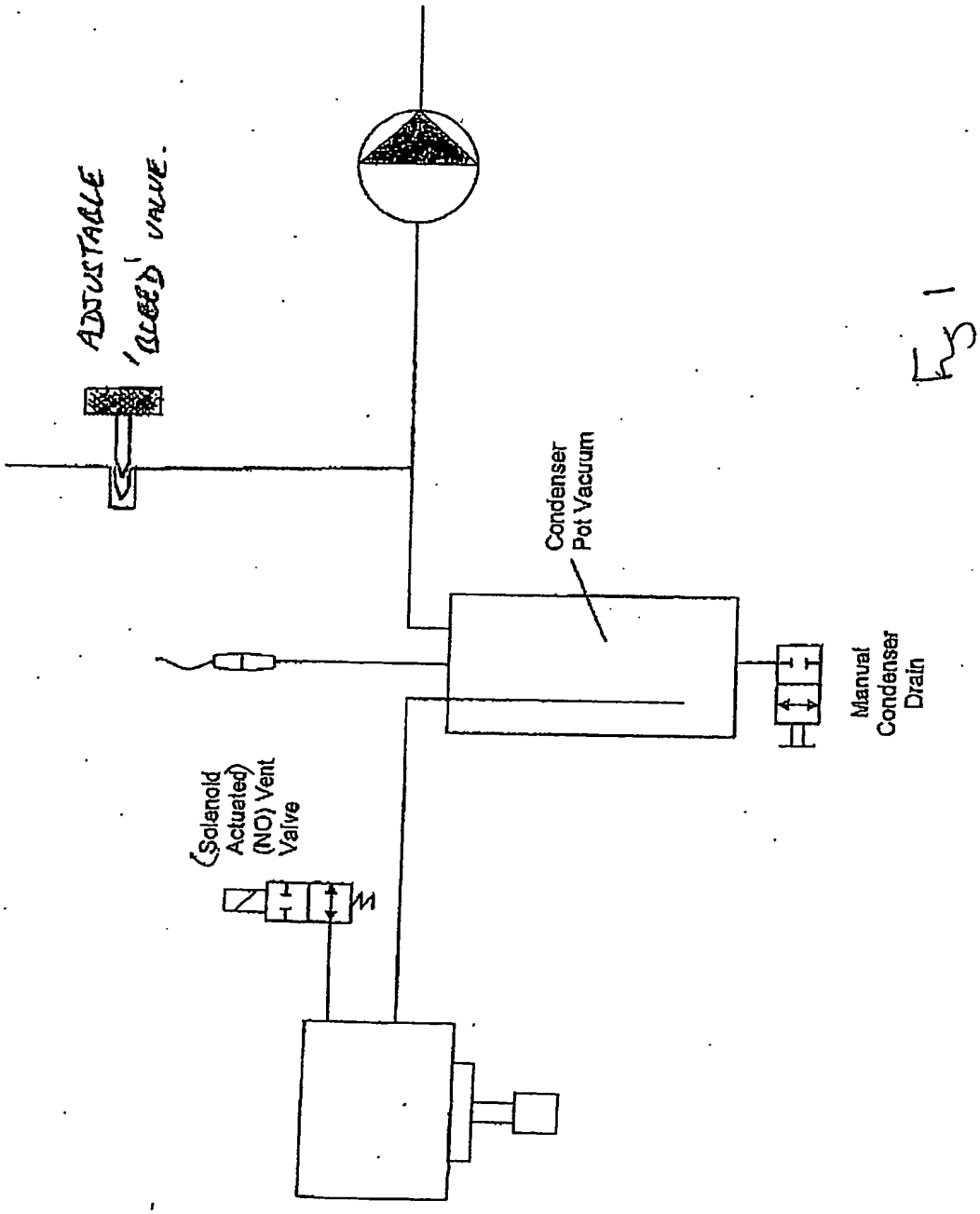
ADJUSTABLE
'BLEED' VALVE.
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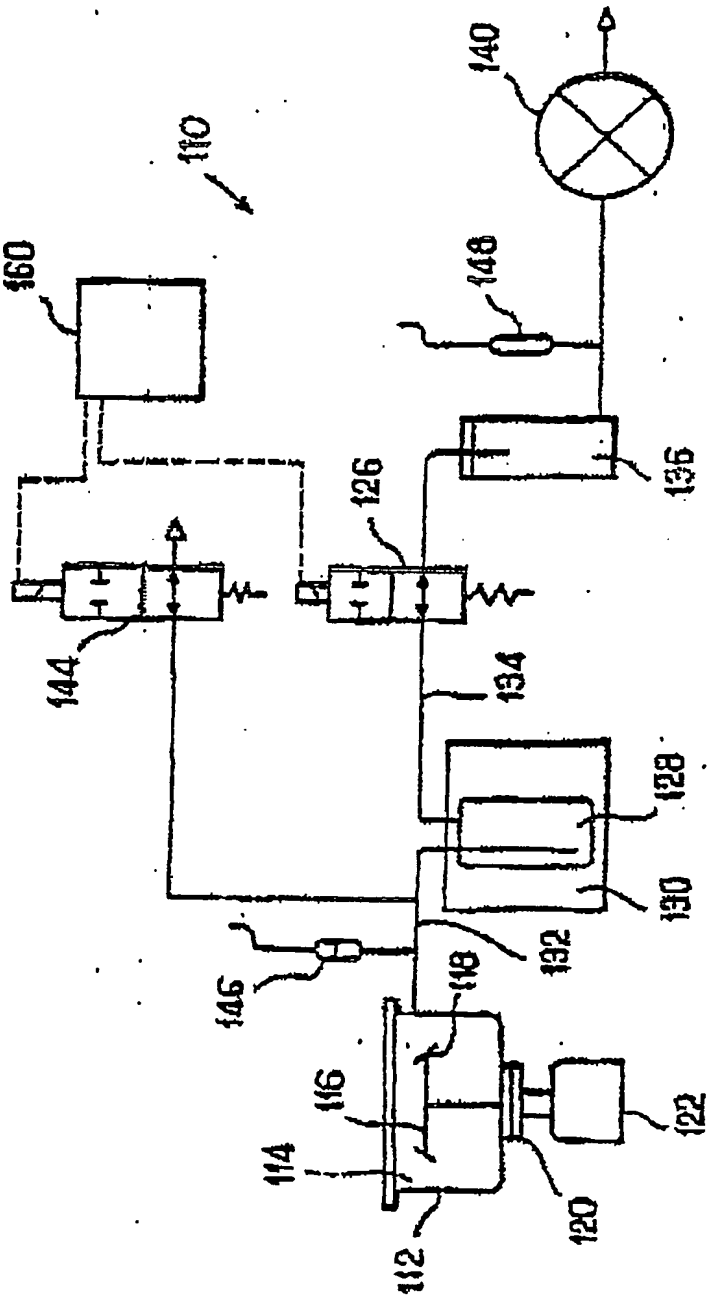
Fig 1

FIG. 2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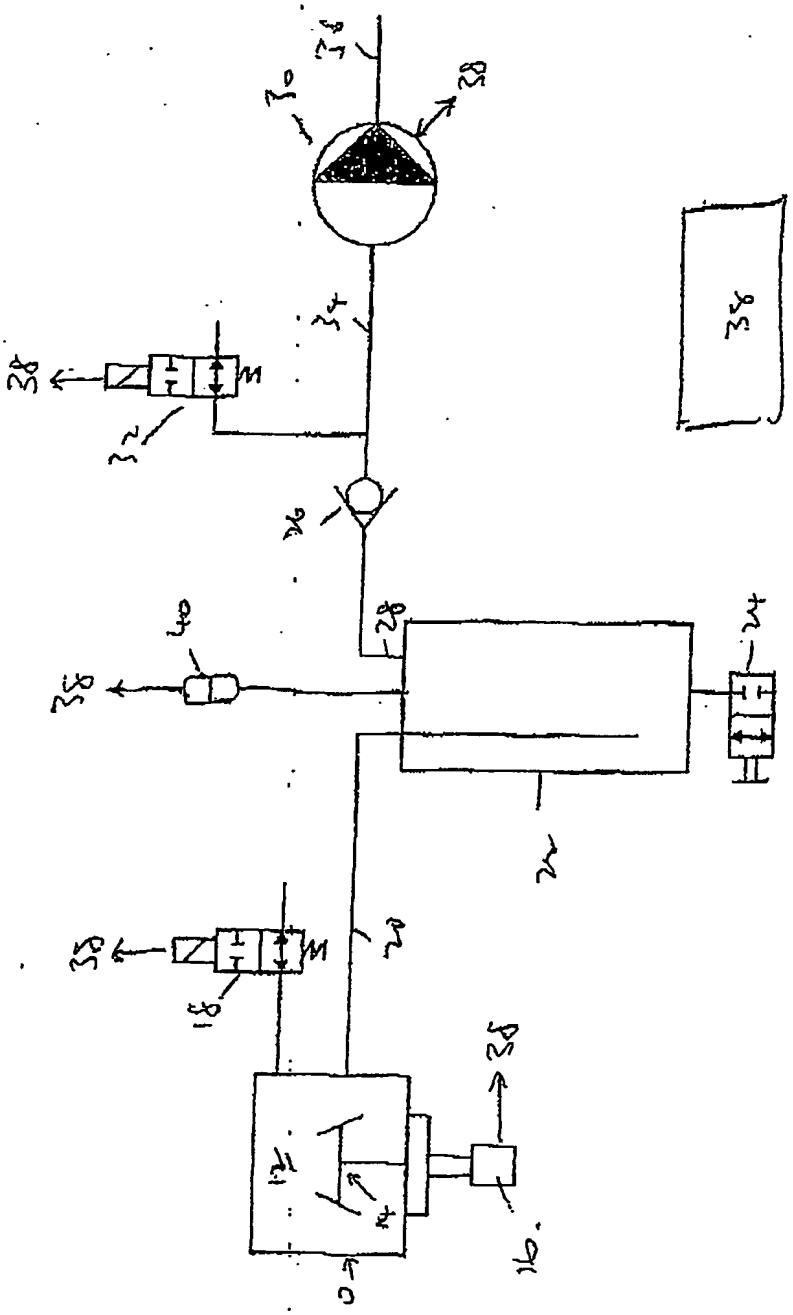

Fig 3